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Electrodeposition of Cuprous Oxide on Boron Doped Diamond Electrodes

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Abstract. *Nowadays, Cu_2O is very promising electrode material for photoelectrochemical applications. In this paper, we report on the controllable synthesis of Cu_2O single particles as well as compact layers on Boron Doped Diamond (BDD) electrodes using potentiostatic deposition in continuous and pulse mode. The BDD layers were prepared with different B/C ratios in the gas phase in order to investigate boron doping level influence on the Cu_2O properties. The effect of electrodeposition conditions such as deposition regime and pulse duration was investigated as well. The Cu_2O covered BDD electrodes were analysed by Scanning Electron Microscopy (SEM) and Raman spectroscopy. Improvement in the homogeneity of the electrodeposit and removal of clusters were achieved when the pulse potentiostatic regime was used. Using the same pulse electrodeposition parameters, we confirmed the possibility of controlling the deposition rate of Cu_2O by varying the BDD conductivity. Finally, we were able to scale the size of Cu_2O particles by changing the number of deposition pulses. The obtained results have shown a great potential of controlling the morphology, amount, size and distribution of Cu_2O films on BDD substrates by changing the boron doping level and electrodeposition conditions as well. The investigations reported herein allowed us to better understand the deposition mechanism of Cu_2O on BDD electrodes which could then be used for preparation of active layers for electrochemical applications and in optoelectronic devices such as solar cells and photodetectors.*

Keywords

Boron doped diamond, cupric oxide, chemical vapor deposition, pulse plating.

1. Introduction

Diamond possesses many exceptional properties such as the highest hardness and thermal conductivity, excellent wear resistance, chemical inertness and the lowest friction coefficient. Therefore, diamond films grown by Chemical Vapor Deposition (CVD) found wide range of applications in electronics, optics, electrochemistry and other fields [1], [2], [3] and [4]. In addition, controlled boron doping enables to adjust conductivity which has a positive impact mainly on the sensitivity and selectivity of BDD electrodes used for electrochemical applications [5]. Electrochemical reactions perform mostly at the interface between electrolyte solutions and the electrodes surfaces. Therefore, modification of the electrode surface morphology is a key factor of tuning the electrode performance [6] and [7]. Electrodeposition of cuprous oxide (Cu_2O) microcrystals may be used to control surface properties for biosensing or biocatalytic applications. Cu_2O is well known as one of the stable oxide forms of copper which has a brownish-red color and a bandgap of

2.0–2.2 eV [8]. Cuprous oxide is an interesting substitute for noble metal catalysts owing to its significant catalytic activity [9]. Other advantages of Cu_2O include the abundance of the precursor materials, nontoxic nature and cost effective synthesis routes. Cu_2O exhibits several attractive characteristics for optoelectronic devices such as solar cells, lasers and photodetectors due to its high absorption coefficient and good majority carrier mobility [10] and [11]. BDD is very promising candidate as a substrate for Cu_2O based photoelectrodes due to chemical inertness, mechanical stability, and it supports the charge separation within the semiconductor and facilitate the photocatalytic activities in general [12] and [13]. Cu_2O thin films can be prepared using various methods including chemical deposition, reactive evaporation, Chemical Vapor Deposition (CVD), thermal oxidation and sol-gel [8]. In contrast to other synthesis processes electrosynthesis is a relatively simple and versatile technique that is performed at atmospheric pressure and does not require high temperatures. Moreover, it allows a good control of the products by controlling parameters such as potential, current densities, pH or precursor concentration in the electrolyte. This work is aimed to demonstrate the possibility of electrodeposition of Cu_2O particles and full layers on the BDD electrodes. For the first time, the influence of boron doping level on the Cu_2O morphology is investigated.

2. Experimental

Polycrystalline BDD electrodes were prepared by plasma enhanced Hot Filament Chemical Vapor Deposition (HF CVD) [14]. Before the BDD deposition the silicon substrates were seeded in an ultrasonic bath in suspension of 50 mg nanodiamond powder (diameter < 10 nm, CAS No. 7782–40–3, Sigma Aldrich) in 1 l of demineralized water (18 MΩ) for 40 minutes. The BDD layers with thickness of 300±50 nm were deposited for 2 hours in a CH_4/H_2 gas mixture with an addition of trimethylboron at process pressure of 3 000 Pa. The B/C concentration in the gas phase was set up to 0, 2 000, 4 000, 8 000, 10 000, 15 000 and 20 000 ppm and the concentration of CH_4 in H_2 to 1 %. The substrate holder temperature 700 °C was monitored during the growth process by a thermocouple. Finally, BDD samples were fixed into printed circuit board support, electrically connected by a silver paste and isolated by a polymer paste (ESL 240-SB) (Fig. 1). Solution containing 100 mM sodium acetate, $C_2H_3NaO_2$, and 100 mM copper (II) acetate, $Cu(CH_3COO)_2$, with pH of 5.6 was used to synthesize the Cu_2O. Electrodeposition of Cu_2O particles was conducted in a glass cell with a BDD working electrode, platinum counter electrode and a silver/silver chloride reference electrode (Ag/AgCl 1 M saturated KCl) using a BioLogic SP–150 galvanostat/potentiostat. In the case of potentiostatic deposition, a potential of −1 V was applied for 10 s and in the case of potentiostatic pulse electrodeposition, 5 pulses of −1 V for 2 s with duty cycle of 50 % were applied. The potentiostatic and pulsed potentiostatic protocols were chosen in order to have similar total charge for both electrodeposition procedures. Prepared series of BDD layers were analyzed by scanning electron microscopy and Raman spectroscopy at room temperature. Scanning electron micrographs of Cu_2O layers were obtained using a JEOL 7500F instrument operating at 10 kV. Micro-Raman spectroscopy (Confocal Raman microscopy MonoVista CRS 750/BX51 manufactured by S&I Spectroscopy) spectroscopy was performed using an argon laser ($\lambda = 514.5$ nm) as the excitation source. Each presented spectrum is an average of twenty continuously acquired spectra with a collection time of 3 seconds each.

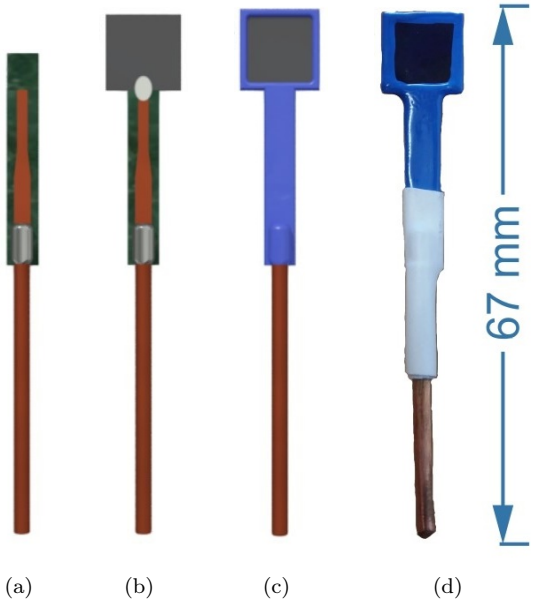

Fig. 1: Process of fixation of BDD electrodes into the electrode holders – (a) electrode holder, (b) connection of BDD electrode using conductive silver paste, (c) applying of polymer isolation paste, (d) photo of real electrode.

3. Results and Discussion

First, we have investigated the difference between pulse and continuous deposition. For this purpose, the BDD electrode with concentration B/C = 10 000 ppm and CH_4/H_2 = 1 % was used (Fig. 3(a)). By applying the pulse deposition (Fig. 2), Cu_2O grains doubled in size compared to the normal potentiostatic deposition were obtained along with the removal of clusters and improved homogeneity of the deposit (Fig. 3(c)). We suppose that it is because each of the pulses initiates a new nucleation processes on the surface site with the

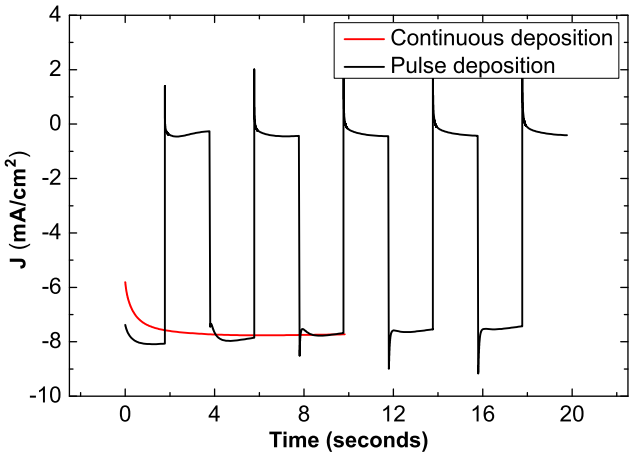

Fig. 2: Evolution of the current density during the continuous and pulse electrodeposition of Cu_2O.

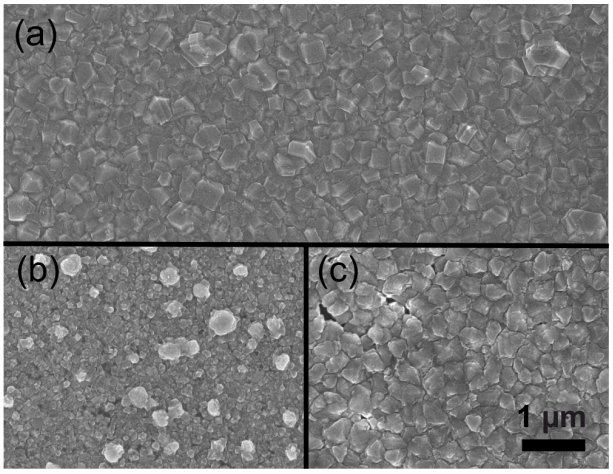

Fig. 3: SEM micrographs showing (a) BDD substrate before Cu_2O deposition, (b) Cu_2O layer deposited by continuous deposition (10 s) and (c) Cu_2O layer deposited by pulse deposition (5×2 s).

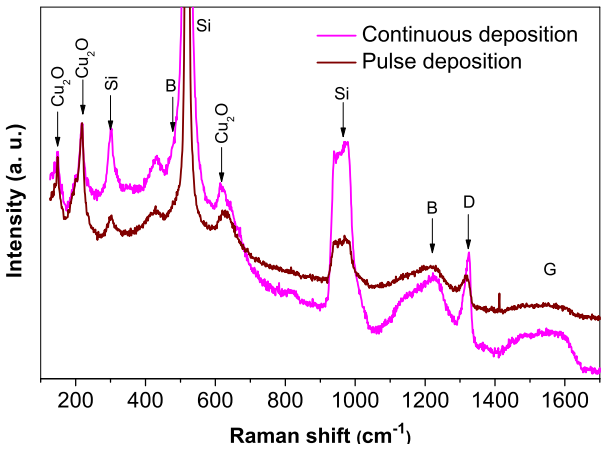

Fig. 4: Raman spectra of Cu_2O deposited on BDD electrodes with B/C = 10 000 ppm and CH_4/H_2 = 1 % using potentiostatic deposition and pulse potentiostatic deposition.

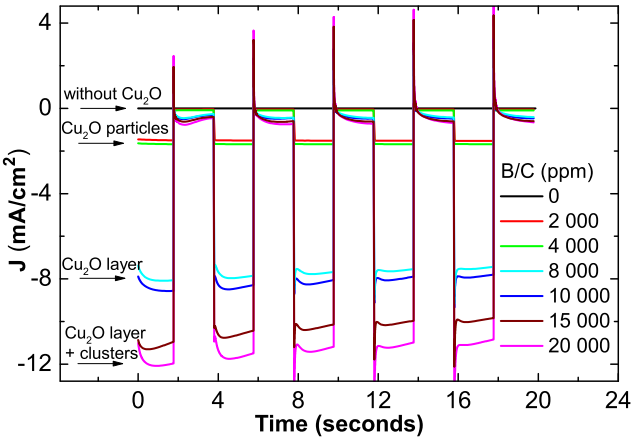

Fig. 5: Evolution of the current densities during the pulse electrodeposition of Cu_2O on BDD substrates prepared by different B/C ratios.

best conditions for electrodeposition which results in a more uniform deposition. This is first represented by the more conductive BDD layer and after a homogeneous covering also by the formed Cu_2O grains. This behaviour causes a more uniform and faster deposition. On the other hand, in the case of continuous deposition sharper Cu_2O crystals were observed, which might be more favourable for electrochemical reactions in some cases (Fig. 3(b)).

The prepared samples of continuously and pulse deposited Cu_2O BDD layers were analyzed by Raman spectroscopy (Fig. 4). The boron doping is represented in Raman spectra by two broad bands at approximately 500 cm^{-1} and 1220 cm^{-1}, which are associated with incorporation of boron into the diamond lattice. The 500 cm^{-1} maximum is attributed to the local vibrational modes of the boron pairs and the small shoulder at 1320 cm^{-1} is associated with polycrystalline diamond in highly boron doped films [15] and [16]. Raman peaks corresponding to Cu_2O are observable at 152 cm^{-1}, 216 cm^{-1} and 630 cm^{-1} [17]. The observed Raman signals indicate the presence of Cu_2O while no CuO peaks were detected. The detected Raman peaks are in good agreement with literature values of Raman modes for Cu_2O nanostructures [8]. The broad maximum at 303 cm^{-1}, sharp peak at 521 cm^{-1} and the broad one at 950 cm^{-1} belong to the silicon substrate [8]. In comparison with a continuous deposition, the pulse deposition exhibits an increase of the Cu_2O signals and a decrease of the other peaks which is most probably caused by a thicker Cu_2O layer.

In the next part, we investigate the influence of boron doping level on the electrodeposition of Cu_2O. Figure 5 shows evolution of the deposition current densities during the pulse electrodeposition of Cu_2O on the BDD samples with various boron doping levels. In our previous study, Svorc et al reported a significant decrease of sheet resistance with increase of boron content

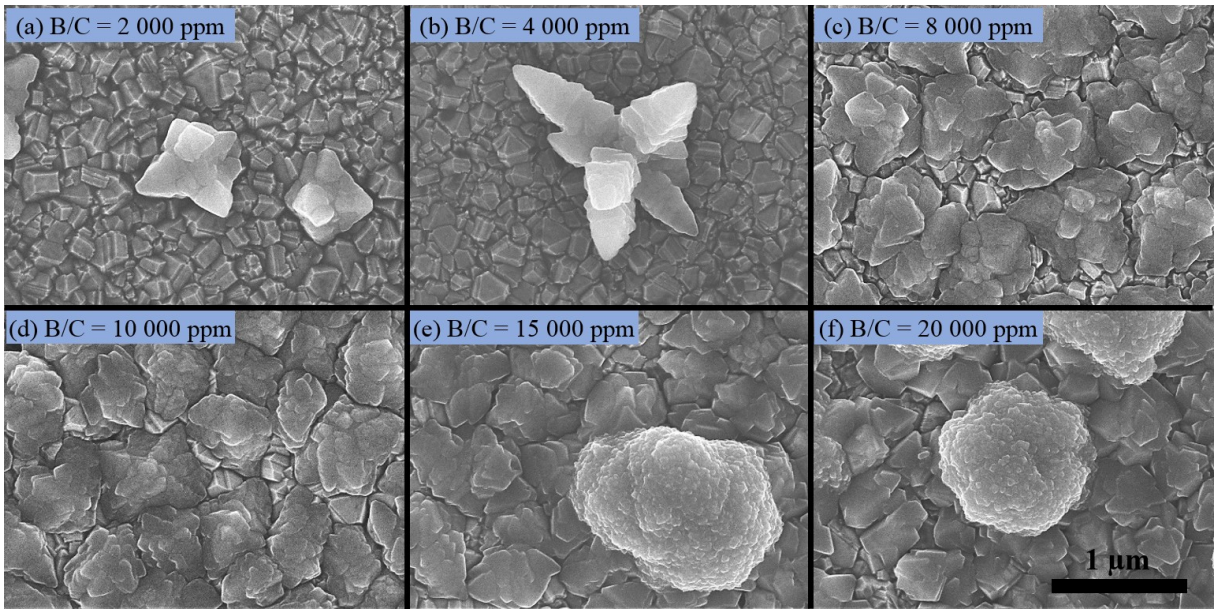

Fig. 6: SEM micrographs showing (a), (b) Cu_2O crystals, (c), (d) Cu_2O layer, (e), (f) Cu_2O layer with clusters.

in diamond [18]. Thus the increase of boron concentration and related higher electrode conductivity and higher amount of surface active sites accelerated the electrodeposition of Cu_2O on the BDD surface. The enhanced BDD conductivity enables an increase of the total electric charge passed through the substance and, following the Faraday's law, a consequent increase of the mass deposited on the electrode surface. In the case of fully covered BDD substrates with Cu_2O layer, 8–11 % decrease of current density was observed during the pulse deposition. This phenomenon is caused by covering the more conductive BDD layer with the less conductive Cu_2O layer.

Figure 6 shows the scanning electron micrographs of Cu_2O crystals deposited on BDD electrodes with various boron concentrations after pulse deposition shown in Fig. 5. On the electrode with B/C = 0 ppm no deposited Cu_2O was observed, which is probably due to very low BDD conductivity and insufficient charge transfer. On the surface of electrodes with B/C = 2 000 ppm and 4 000 ppm, homogenously distributed Cu_2O crystals with diameters in the range of 1–3 μm were observed. The electrodes with B/C = 8 000 and 10 000 ppm were almost continuously covered by Cu_2O layer, and on the electrodes with ratio B/C = 15 000 ppm and higher, the covering was even denser and spherical clusters on top of a compact Cu_2O layer were observed in addition. These results indicate a direct influence of the electrode conductivity determined by boron doping level on the total current density and consequent mass transport, thus influencing density and morphology of Cu_2O on the BDD surface providing an effective tool of surface properties controlling. The Raman spectra (Fig. 7) show increased Cu_2O peaks at 152 cm^{-1} and at 216 cm^{-1} in the case of electrodes with higher boron doping level [8].

This indicates the presence of thicker layers caused by faster growth on more conductive BDD substrate. The increase in intensity of 500 cm^{-1} and 1 220 cm^{-1} maxima follows the increase in B/C ratio in the gas mixture during deposition of BDD films evidencing the increasing doping level. A peak at 1 332 cm^{-1} corresponding to the polycrystalline diamond was also recorded in spectra of the films with lower and no boron concentrations [15].

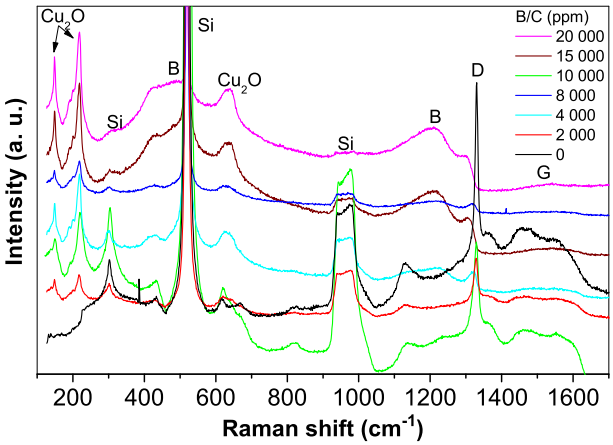

Fig. 7: Raman spectra of Cu_2O deposited on BDD electrodes with various B/C concentration and CH_4/H_2 = 1 % using pulse deposition.

Finally, we tried to control the size of the Cu_2O layer grains by varying the duration and number of the pulses maintaining the same charge. For this purpose, we choose the electrode with

B/C = 8 000 ppm where no Cu_2O clusters were observed. Three different depositions consisting of (1) five pulses with a duration of 400 ms and duty cycle of 10 % (Fig. 8(a)), (2) ten pulses with a duration of 200 ms and duty cycle of 5 % (Fig. 8(b)), and (3) twenty pulses with a duration of 100 ms duty cycle of 2.5 % (Fig. 8(c)) were performed. SEM micrographs have shown that the size of Cu_2O grains was successfully scaled down by applying higher number of pulses with shorter duration. This phenomenon is caused by a termination of particle growth and nucleation of new particles during next pulse. Similar influence of deposition pulses on size of Cu_2O particles was reported by Liau et al [19]. Based on the experimental results and theoretical knowledge we can assume that the number and size of grains can be directly affected by the number and duration of the pulses, which together with the boron doping level give a great potential to control the amount, size and distribution of Cu_2O on BDD substrate.

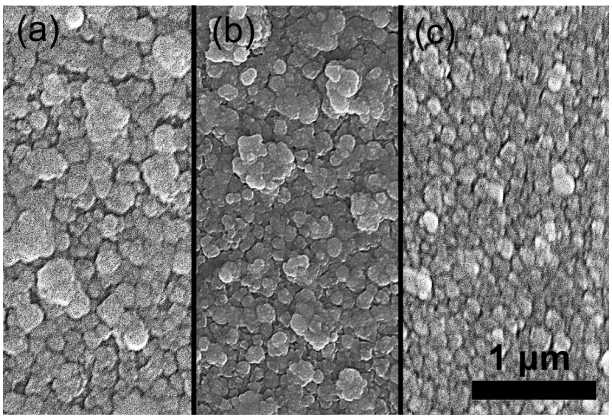

Fig. 8: Morphology of Cu_2O layers deposited using different pulses durations. The duration was set up to (a) 400 ms, (b) 200 ms and (c) 100 ms.

4. Conclusion

In this study, we demonstrated the possibility of controllable electrodeposition of Cu_2O particles as well as compact layers on the boron doped diamond electrodes. The BDD electrodes fabricated with various B/C concentration in gas mixture (0–20 000 ppm) were used for the depositions of Cu_2O by potentiostatic pulse electrodeposition. Comparison of continuous electrodeposition with pulse electrodeposition showed significant enhancement of the homogeneity using a pulse electrodeposition. The investigations of boron doping level influence on the morphology of Cu_2O on BDD substrate has shown several interesting results. In the case of non doped diamond, no Cu_2O deposition was observed. In the case of B/C = 2 000–4 000 ppm, the Cu_2O micro particles were observed and in the case of B/C > 8 000 ppm the BDD the surface was fully covered with a Cu_2O layer. The obtained results indicate a significant influence of the B/C ratio on the deposition rate, density and the morphology of Cu_2O films caused by different BDD conductivity and surface-active sites distribution. Following the SEM investigation, we also found that the electrodeposited layers of Cu_2O particles were homogeneously distributed over the whole BDD substrate. Finally, we successfully controlled the size of the Cu_2O grains by varying the number and duration of the deposition pulses. The presented investigations have shown a great potential of controlling the morphology, amount, size and distribution of Cu_2O films on BDD substrates through the change of boron doping level and electrodeposition conditions as well.

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